OBSERVATIONS OF THE EFFECTS OF LOADING DENSITY ON THE INITIATION AND GROWTH OF DETONATION IN AZIDES

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OBSERVATIONS OF THE EFFECTS OF LOADING DENSITY ON THE INITIATION AND GROWTH OF DETONATION IN AZIDES

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ABSTRACT: The ease with which lead azide builds from burning to detonation was measured for a number of different lots obtained from a variety of sources. The hot wire sensitivity of these various lead azides was also measured. The measurements show that dextrinated lead azide as presently specified is very variable in its build up to detonation properties. Some lots of lead azide are particularly poor in making the transition from burning to detonation at commonly used loading pressures. The hot wire sensitivity of various lead azides is shown to increase with increased density and approach the sensitivity of the most sensitive hot wire explosives.

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This report gives some of the more important and significant results of an investigation which was carried on, with many interruptions for work of more immediate urgency, during 1955, 1956, and 1957 by members of the then Explosive Properties Division, Explosives Department, Naval Ordnance Laboratory.

The experimental work was carried on as part of the effort to gain sufficient understanding of the behavior of primary explosives to make reliable predictions of the performance of primers, detonators, etc. At the time this work was started, the inadequacy of the existing lead azide specification (which was essentially a consolidation of a 1943 Army Specification and a 1941 Navy, Bu. Ord. O.S.) was apparent to most users of lead azide in ordnance. The work reported herein was part of an effort in which the Naval Ordnance Laboratory collaborated informally with Picatinny Arsenal to improve this specification. The overall effort has led to several modifications of the specification in recent years and more may be anticipated. In addition, this material is directly applicable to the design of initiators. The implications of the data presented are believed to be of more than passing significance with respect to ordnance applications of lead azide.

The U. S. Government by this report intends neither to indorse nor criticize any commercial article referred to herein.

W. D. COLEMAN
Captain, USN
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By direction

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OBSERVATIONS OF THE EFFECTS OF LOADING DENSITY ON THE INITIATION AND GROWTH OF DETONATION IN AZIDES

INTRODUCTION

1. Among commonly used explosives, lead azide is unique in the rapidity with which it makes the transition from burning to detonation. Although this is the property for which it is most valued, the military specification covering lead azide includes no quantitative measurement of this property. The only specified test which gives an indication of the growth of detonation, the sand test, is so insensitive to variations in this property as to "pass" a 50% mixture of lead azide with dextrin. (The sand test has been eliminated from recent documentation, PA-PD-1217 (Rev. 1).)

2. If the use of lead azide in ordnance were confined to applications as tolerant of variations as the sand test, the fact that all material made so greatly exceeds specification requirements could be taken as grounds for complacency. However, the development of ordnance has taken a very different course. Under pressure for miniaturization, the charge of lead azide in many military detonators has been reduced to the essential minimum, and at the same time the loading pressure has been greatly increased. Although these changes are desirable for miniaturization, they reduce the tolerable range of material or manufacturing variables in the production of detonators.

3. The choice of loading pressure or density for a detonator is particularly difficult. Although the output and effectiveness of ideal detonation increases with loading density, the voids which are present in explosives as they are loaded, have an important role in the growth of detonation. As the density of an explosive approaches its crystal density, its ability to make the transition from burning to detonation is reduced. Although this trend is relatively gradual (1), the transition from burning to detonation, under any given set of conditions, is usually quite abrupt (2), (3), and (4). Thus, in a given detonator fired by a given stimulus under a given set of confinement conditions, the output often drops quite abruptly when the loading pressure or density is increased above a certain point. This point has been referred to as the "dead pressing" point. Mercury fulminate, which was the standard primary explosive for many years, is particularly easy to "dead press" for conditions of use in fuzes. Because lead azide is so much

less susceptible to this phenomenon than mercury fulminate with which it was compared when first used, it has been stated that lead azide cannot be "dead pressed". Also, probably on the basis of comparisons with mercury fulminate and lead styphnate, it has been stated that lead azide always detonated immediately upon initiation. Neither of these statements is true except in a relative sense. The decrease in output of detonators with increasing loading density observed by Graff and Kabik (5) probably is attributable to the retarded growth of detonation of the lead azide intermediate charges. At very high loading densities and with mild initiation, dextrinated lead azide has been observed to detonate for inches at velocities in the range of 1400 to 1700 meters per second, about one third of its ideal stable velocity (6). Although these effects were observed in material which had been loaded at pressures far beyond those used in ordnance, the possibility that they might be significant at lower pressures for some lots of azide, as used in some items cannot be ignored.

4. In the development of a new detonator, it is customary to perform exhaustive tests to establish that it will perform its function reliably. In spite of such development programs, detonators made in accordance with all drawings and manufacturing specifications sometimes fail either in use or in performance tests. Some of these failures led to the suspicion that detonation growth rates might vary not only with such intentional variation in manufacturing procedures as are used to produce colloidal and PVA lead azide, but also with unintentional lot to lot variations. ("PVA" - polyvinyl alcohol).

5. With the motivation of this suspicion, samples were collected from nine installations of twenty-four lots of lead azide which were being used to load ordnance and experimental material. These twenty-four lots of lead azide as well as two lots of silver azide which happened to be on hand were subjected to a series of tests. The largest part of the effort was expended in the comparison of the growth of detonation in these samples. However, some of the other information obtained in the course of this investigation may be of equal interest.

THE GROWTH OF DETONATION AS MEASURED BY THE DENT TEST

6. Previous work (7) indicated that the depth of the dent in a steel plate which results from the detonation of a column of explosive is very nearly proportional to the length of the column for column lengths less than the diameter of the column. For longer lengths, the increase of dent with column length began to level off due to radial losses. Other experiments had shown that the depth of this dent was proportional to the product of the detonation pressure and the length of the detonation head (8).

7. The measurements which led to these conclusions had been carried out under conditions for which stable detonation was anticipated and apparently realized. The results encouraged the belief that the dent test could be used as a means for observing the growth of detonation. Savitt (9) used dent tests to observe the growth of detonation in secondary explosives. In this application, the relatively vigorous initiation needed tends to complicate interpretation. In contrast, for primary explosives, in which detonation may grow from gentle burning, quite accurate estimates are possible of the exact column length required to effect this transition. If, as in Figure 1, the depth of dent is plotted versus the column length for short enough columns, it is found that the relationship is nonlinear in this range. This nonlinearity may be interpreted, in terms of the relationships mentioned above, as an indication of the growth pattern of detonation. The zero intercept, for example, may be taken as the length of column required to build to detonation and the slope considered to be proportional to detonation pressure. This interpretation is quite clearly oversimplified in view of the fact that the column length in these experiments is much shorter than its diameter. The sigmoid shape of some of the curves of Figure 1 is more reasonably explained in terms of growth and propagation in three dimensions than in one. Since these explanations are involved, tentative, and somewhat academic at this point, they will be left for future consideration.

STANDARD MICROSCALE PLATE DENT TESTS

8. Arrangement and Procedure. Although it would have been interesting to carry out experiments such as those whose results are plotted in Figure 1 for each of the twenty-three samples, loaded at a series of loading pressures, the number of trials in such a program seemed prohibitive. The data of Figure 1 as well as other preliminary experiments left no doubt that loading density is an important factor in the growth of detonation. Data at hand indicated that the dent produced by a small column of explosive of a given weight would be proportional to the fraction of the material which actually detonated times the detonation pressure. Of more importance to practical detonator design, the depth of dent had been related to the effectiveness of a charge in initiating a subsequent charge of less sensitive material. These considerations led to the evolution of the experimental arrangement and procedure described in the following paragraph.

9. The arrangement for each trial is illustrated schematically in Figure 2. A small sheet or wafer of aluminum was centrally drilled and loaded with a specimen of lead azide. The wafer was placed on a dent block of annealed and ground tool

steel. (Tool steel was used because it was commercially available in the form of annealed and ground flats.) The bend of a hairpin-shaped length of nichrome wire was placed in firm but gentle contact with the top of the azide specimen. Although the force of contact was not measured, it may be estimated, from the design of the apparatus, to have been about two or three grams. The wire was heated by the application of a steady current until the specimen exploded. The depth of the dent was measured by means of a dial indicator gage graduated in ten-thousandths of an inch.

10. Two series of such tests were carried out. In both, the dent block had dimensions of 1" x 1" x 1/4" and the heater wire was ten mils in diameter. In the first series an eight milligram specimen of explosive was loaded into an 0".075 diameter hole in each 0".042 thick wafer of aluminum at seven loading pressures ranging in geometric progression from 2000 psi to 128,000 psi. Three replicate specimens were loaded from each sample at each loading pressure. In the second series, a twenty milligram specimen from each sample of explosive was loaded at each of twenty pressures in geometric progression between 1600 psi and 125,000 psi into a 0".100 diameter hole in a 1/16" thick aluminum wafer.

11. Results. Typical data obtained in these experiments are plotted in Figure 3. Each curve of Figure 3 is plotted from averaged data from several lots for which the data were so similar as to be statistically homogeneous. The data for the lots of dextrinated lead azide seemed to fall into two different groups as shown by the curves labelled Dex A and Dex B on Figure 3.

12. If practically all of a specimen such as used in these experiments were to detonate high order, the depth of dent should increase monotonically with loading pressure, since the detonation pressure increases with loading density and thus with loading pressure. Apparently PVA and colloidal lead azide, in general, behave in this manner.

13. The variation of dent depth with loading pressure in dextrinated lead azide (the curves marked Dex in Figure 3) is quite different. Note that the dent for 100,000 psi is a small fraction of that for material loaded at 10,000 psi. More seriously, note that though the output of some lots is close to its maximum when pressed at 20,000 psi, a very commonly used value for detonators, the output for other lots is approaching its maximum when pressed at this same pressure. An interesting observation, Figure 4, is that a 50/50 mixture of dextrinated and PVA lead azide gave data which scattered between that of its components.

14. A curve such as those in Figure 3 characterizes important aspects of the performance of the explosive to which it refers. For purposes of comparison and categorization, however, numbers are often more useful than plots. Several numerical quantities,

each with its particular significance from an application point of view, may be derived from this type of data. Such numbers include, maximum dent, the loading pressure at which the maximum dent was obtained, the "dead pressing" point, the minimum dent for a specified range of loading pressure, the dent produced at one specific pressure, and the pressure range over which the dent is at least some specified fraction of its maximum. Some of the results of the investigation are condensed in such terms in Table I.

15. Referring to Table I, it will be noted that dextrinated lead azide is quite variable in performance. By each of the criteria used, its proportional variation is greater than that for the PVA lead azide, the colloidal lead azide and the silver azide if the latter are taken as a group. Moreover, note that the dextrinated material is so inferior in output as to overlap the range of the other materials by only two criteria. Perhaps the most disquieting aspect is that the "dead press" point for some lots falls close to the bottom of the range of commonly used loading pressures.

16. Each sample was analyzed chemically.[11] Among the various lots of dextrinated lead azide, no correlation was apparent between lead azide content and performance (Table II). The more vigorous and uniform performance of the other types of azide might be attributable of their higher purity. PVA lead azide had been purposely manufactured with a purity of only 90%, the bottom of the range of the dextrinated material. This low purity lot of PVA lead azide was not noticeably different from the other lots of PVA lead azide, thus other factors must apply.

17. An effort was made to measure the size of the individual crystallites of the agglomerate grains of the dextrinated lead azide. The "spread" of the peaks of x-ray diffraction patterns as measured by means of a goniometer was the first criterion applied. Crystals of smaller sizes have broader peaks because of the aberrations introduced by surfaces. However, the PVA lead azide, of which each grain was quite apparently a single crystal had generally broader peaks than did the dextrinated lead azide (which had much smaller crystals). This spread of the PVA diffraction pattern was attributed to strains of the crystals (10). In any case, it made the PVA useless as a standard for the calibration of the relationship between peak breadth and crystal size and aroused the suspicion that crystal strain might be a dominant factor in the spread of the peaks of the spectra from dextrinated lead azide. The "spottiness" of x-ray powder diffraction patterns was the second criterion applied. The larger crystals, of course, would give spottier patterns because the orientation of each crystal is quite specific. This criterion is qualitative and somewhat subjective.

The subjective aspect was alleviated by having a set of powder photographs sorted in order of spottiness by three persons working independently. The ordering arrived at by the three sorters was in general agreement. No significant correlation between crystallite size and the microscale dent test was obtained. Of course, the PVA lead azide, which had larger crystals, also performed better in the dent test, but this is probably related to other aspects than crystal size. In Figure 5 microscale dent test data for dextrinated and PVA lead azide as received are compared with similar data for the same materials which had been ball milled. The improvement in output of the dextrinated material with milling may be attributable to either the reduction of particle size or the exposure of surfaces uncoated with dextrin. The reduction of output of the PVA lead azide with milling is quantitatively attributable to the reduced loading density attained at each loading pressure.

18. The microscale dent test data for dextrinated lead azide, close to the dead pressing point, shows appreciable scatter. The data plotted as crosses in Figure 6 are an example. This scatter might be attributed to: (a) the randomness of the growth of detonation, (b) variability of the pressing properties of the explosive, (c) variation in effective loading pressure due to buckling and binding of the small ram used, and (d) weighing errors. The relatively large variation in the measured column heights is evidence that at least one of the last three of these sources of variation is present. The circles in Figure 6 are the same data plotted versus column length. (The relationship between column length and pressure as indicated in Figure 6 was obtained by averaging and smoothing data from various measurements.) Note that the circles are more closely grouped about the solid curve obtained in previous measurements than the crosses, indicating at least some degree of correlation between column length and dent depth. Although this does not eliminate the possibility that some of the scatter is due to variability in the detonation growth process (factor (a)), it does indicate that one or more of the other factors are involved. The fact that the slope of the best curve through the circles is so much steeper than the dashed line obtained from data in which the column length was varied by purposely varying the quantity of explosive used indicates that factors other than weighing errors are important. The data at hand offer no means of differentiating between factors (b) and (c). It is possible, then, that the scatter in microscale dent test data is attributable to variability of pressing properties of dextrinated lead azide within each lot.

HOT WIRE SENSITIVITY

19. Where short and reproducible response times are required, lead azide has found increasing application as a flash charge material for electric initiators. Moreover, hot wire sensitivity had formed a part of the NOL primary explosive evaluation program. The usual practice was to load the material at about 4000 psi against the surface of a flush bridged initiator plug.

20. The physicist who was carrying on the program discussed herein was relatively new to the initiator field. Not knowing that it was impractical to load explosives against a bridgewire at more than ten or twenty thousand pounds per square inch, he issued loading orders for test initiators loaded at 3300, 10,000, 30,000, and 90,000 psi. The bridgewires broke in about half of the initiators loaded at 90,000 psi, so the ordnancemen, though somewhat perturbed, loaded twice as many as called for.

21. The initiators used were bridged with tungsten wire 0.27 mils in diameter by 30 mils long. About twenty-five to thirty were loaded with azide* from each sample at each of the loading pressures mentioned. They were tested using the Bruceton up-and-down technique at a constant twenty-four volt discharge from condensers varying in capacitance with logarithmic steps.

22. The results of these tests, shown in Figure 7, were quite surprising. Note that the energy requirement decreases with increasing loading pressure. The mean firing energies of the various azides, when pressed at 90,000 psi, fall in the range of the most sensitive flash charge materials used.

CONCLUSIONS

23. The following conclusions may be drawn.

a. The specification for lead azide should include a means of evaluating its detonation growth rate.

b. Dextrinated lead azide which passes the present specifications is quite variable in this respect.

*Azides were milled 24 hours in a ball mill before loading in hot wire sensitivity test.

c. For applications involving small charges of azide, where detonation is required, azides other than dextrinated lead azide are preferable.

d. In initiators of sufficiently rugged design to be loaded at pressures in the range of 90,000 psi, both lead and silver azide may offer substantial advantages worth the solution of the problems which attend such loading.

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Table I
Microscale Dent Data for Various Azides

	Specimen Wt. (mg)	Lead Azide Dextrinated (d)	Lead Azide PVA (p)	Lead Azide Colloidal (c)	Silver Azide (two lots)
Maximum dent	8	0.7-1.6 mils	2.2-3.2 mils	2.5 mils	3.1-4.1 mils
	20	2.0-4.7 mils	4.7-5.9 mils	4.8 mils	6.7-7.2 mils
Dent at 10,000psi	8	0.4-1.6 mils	1.7-2.6 mils	2.2 mils	2.3-2.6 mils
	20	1.1-3.7 mils	3.4-4.5 mils	4.1 mils	4.7-5.9 mils
Dent at 40,000psi	8	0.0-0.5 mils	2.0-2.9 mils	2.8 mils	3.1-3.5 mils
	20	0.05-4.6 mils	5.4-6.6 mils	4.8 mils	5.6-6.8 mils
Minimum dent (4000-16000 psi)	8	0.05-0.9 mils	1.1-1.9 mils	1.8 mils	1.7-1.8 mils
	20	0.5-2.6 mils	2.7-3.4 mils	3.4 mils	3.1-4.9 mils
Minimum dent (16000-64000 psi)	8	0.0-0.5 mils	1.7-3.3 mils	2.3 mils	1.9-2.6 mils
	20	0.0-1.8 mils	4.5-4.9 mils	4.0 mils	4.6-6.0 mils
"Dead Press" point (a)	8	10.6-35 kpsi	80->130 kpsi	>130 kpsi	85->130 kpsi
	20	11.7-128 kpsi	>130 kpsi	>130 kpsi	73->130 kpsi

(a) The "dead press" point is defined for this purpose as the loading pressure (greater than that for maximum dent) at which the dent value has fallen to 1/2 its maximum value.
(d) Range of data from sixteen lots of dextrinated lead azide, each of which had passed MIL-L-3055
(p) Range of data from six lots of polyvinyl alcohol lead azide made by the Olin-Mathiesen Chemical Company by their patented process.
(c) Colloidal lead azide made by DuPont by a process developed at Picatinny Arsenal.

Table II
Microscale Dent Test Results for Individual Lots of Explosives*
(Test Conditions: 0.075 inch dia. 8 mg charges)

Lot Iden.	Type	Mfr	(Ref.(11)) Purity (%)	Min. Dent (a) (mils)	3rd High Dent (mils)	Dent for 10 kpsi (mils)	Pressure for Max. Dent (kpsi)	"Dead Press Point"(b) (kpsi)
X233	Dex	A	93.5	0.0	0.6	0.4	6.4	11
X232	Dex	A	92.2	0.2	0.8	0.9	10	13
X226	Dex	B	93.4	0.6	1.0	0.6	5-8	11
X243	Dex	A	91.6	0.5	0.7	0.6	8	18
X227	Dex	C	91.4	0.5	0.8	0.8	8	17
X234	Dex	A	93.4	0.7	1.0	0.9	5-12.5	14
X230	Dex	A	93.4	0.5	1.3	1.0	8	10.8
X236	Dex Col	C	90.0	0.3	1.2	0.7	20	36
X225	Dex	B	93.4	0.7	1.1	1.1	6.4-8	14
X239	Dex	C	93.1	0.6	1.2	1.1	6.4-8	14
X235	Dex	C	93.9	1.0	1.1	1.1	4-10	14
L41	Dex	D	92.3	0.7	1.1	1.1	8	34
X240	Dex	D	92.3	0.8	1.3	1.0	4	25
X231	Dex	A	92.6	0.9	1.2	1.2	8	18
X237	{Dex 50 PVA 50	C	93.4	1.1	2.3	1.3	100	45-90 (c)
X201	Ag	C	99.4	1.6	2.9	2.2	45	90
X228	PVA	C	95.1	2.1	2.4	2.1	64	125
X238	PVA	C	90.0	2.0	2.5	1.7	50	>125
X242	PVA	C	94.1	2.2	2.5	1.8	64	>125
X114	PVA	E	~95.0	2.0	3.1	2.3	32	74-125+ (c)
X229	Col	A	98.9	2.3	2.9	2.1	125	>125
X169	PVA	C	~95.0	2.2	3.7	2.6	125	40-125+ (c)
X241	PVA	C	95.6	2.8	3.4	2.2	100	>125
X117	Ag	C	99.1	2.7	3.8	2.4	64	120

*Arranged in ascending order of "brisance" and resistance to "dead pressing".

(a) Minimum dent within the loading pressure range of a factor of four for which this minimum is largest.

(b) The "dead press point" was considered to be that at which a plot of depth of dent versus logarithm of loading pressure descended to half of its peak.

Dex - Dextrinated Lead Azide (MIL-L-3505)
Col - Colloidal Lead Azide
PVA - Polyvinyl Alcohol Lead Azide (proprietary Olin-Mathiesen)
Ag - Silver Azide

(c) Relation between dent and loading pressure was bimodal.

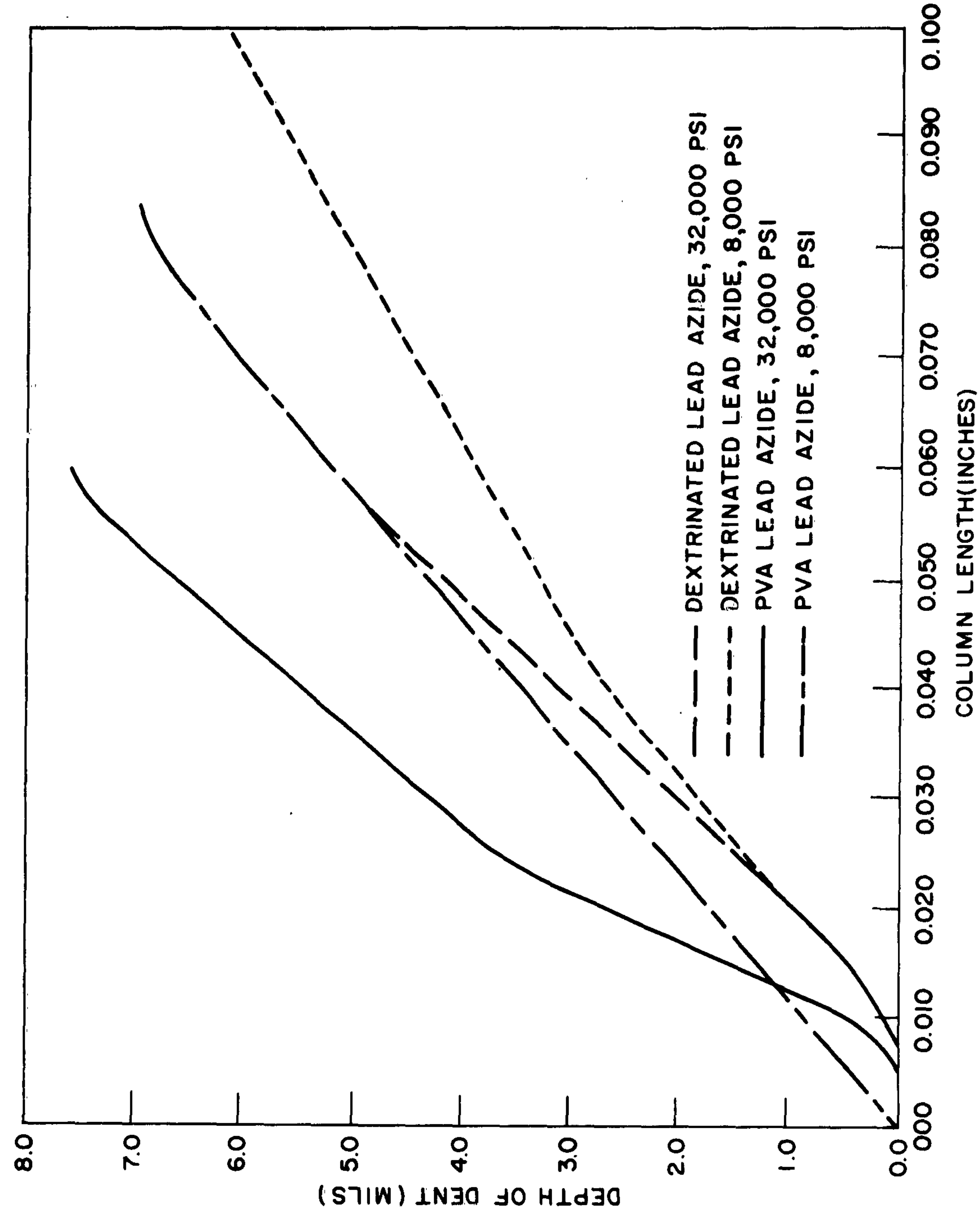

FIG. I DEPTH OF DENT VS COLUMN LENGTH FOR SHORT COLUMNS OF AZIDE

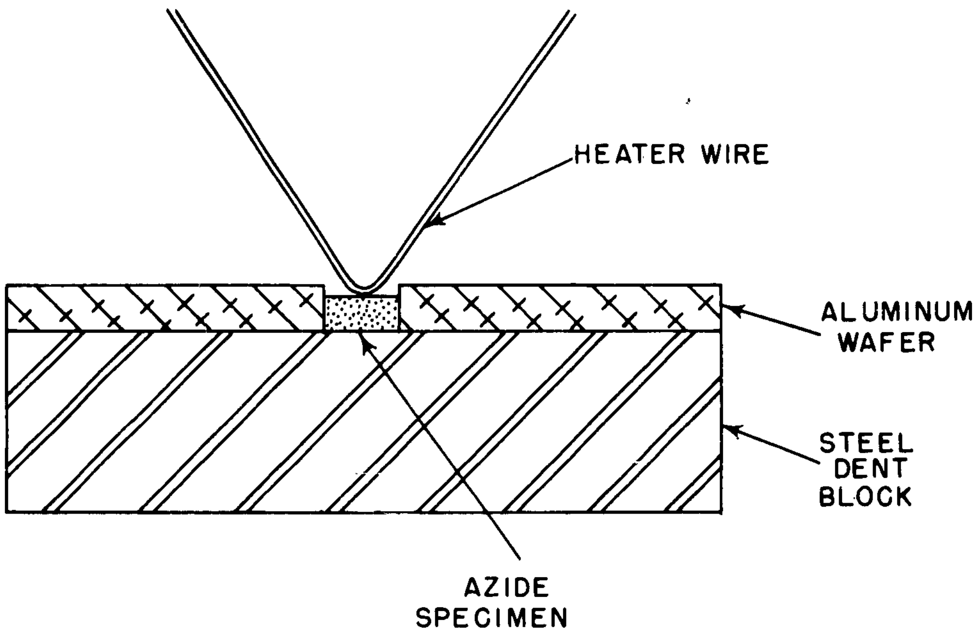

FIG. 2 EXPERIMENTAL ARRANGEMENT FOR MICROSCALE PLATE DENT TEST

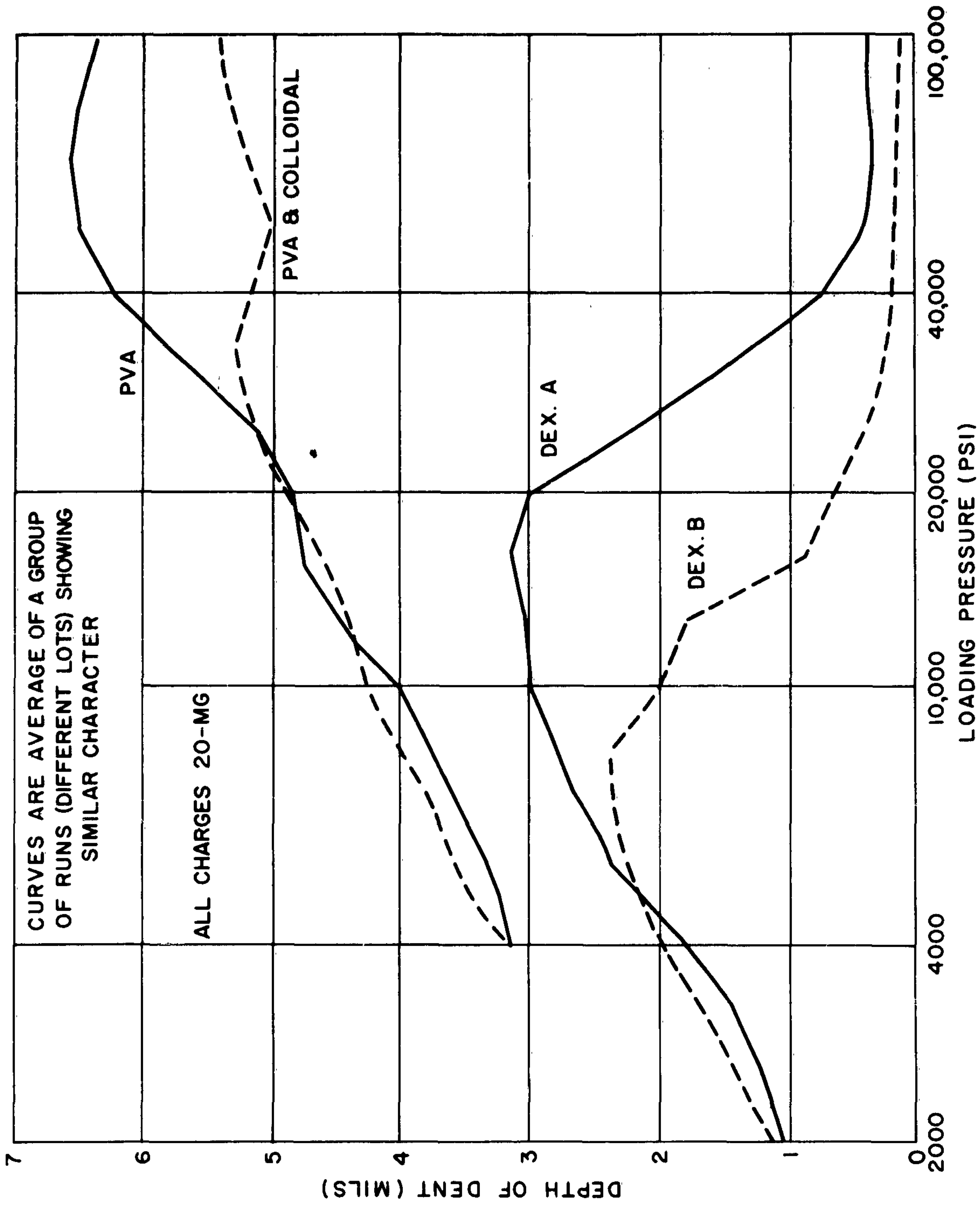

FIG. 3 STEEL PLATE DENT TEST, MICROSCALE

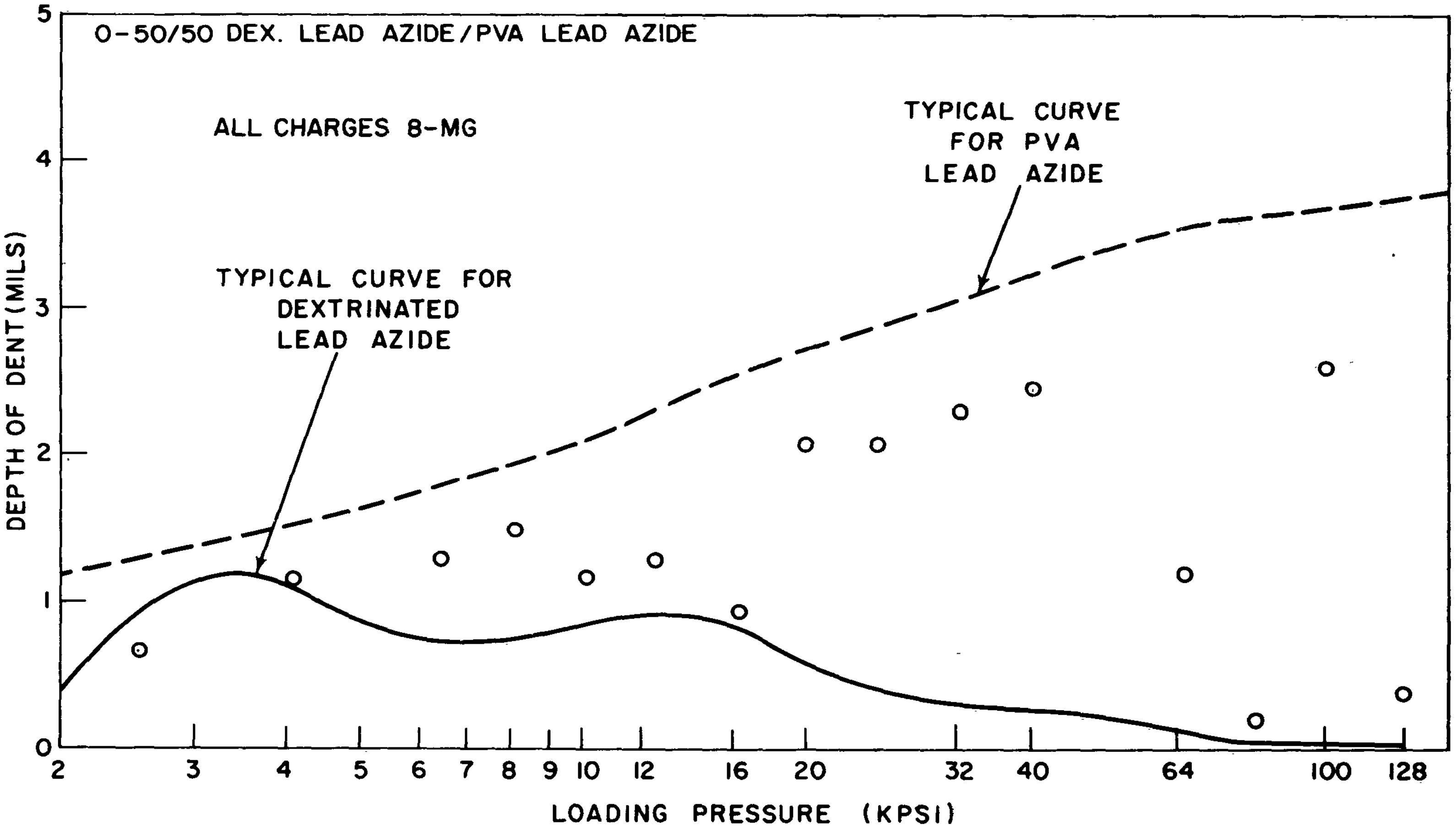

FIG. 4 MICROSCALE DENT FOR DEXTRINATED LEAD AZIDE, PVA LEAD AZIDE AND A 50/50 MIXTURE OF DEXTRINATED WITH PVA LEAD AZIDE

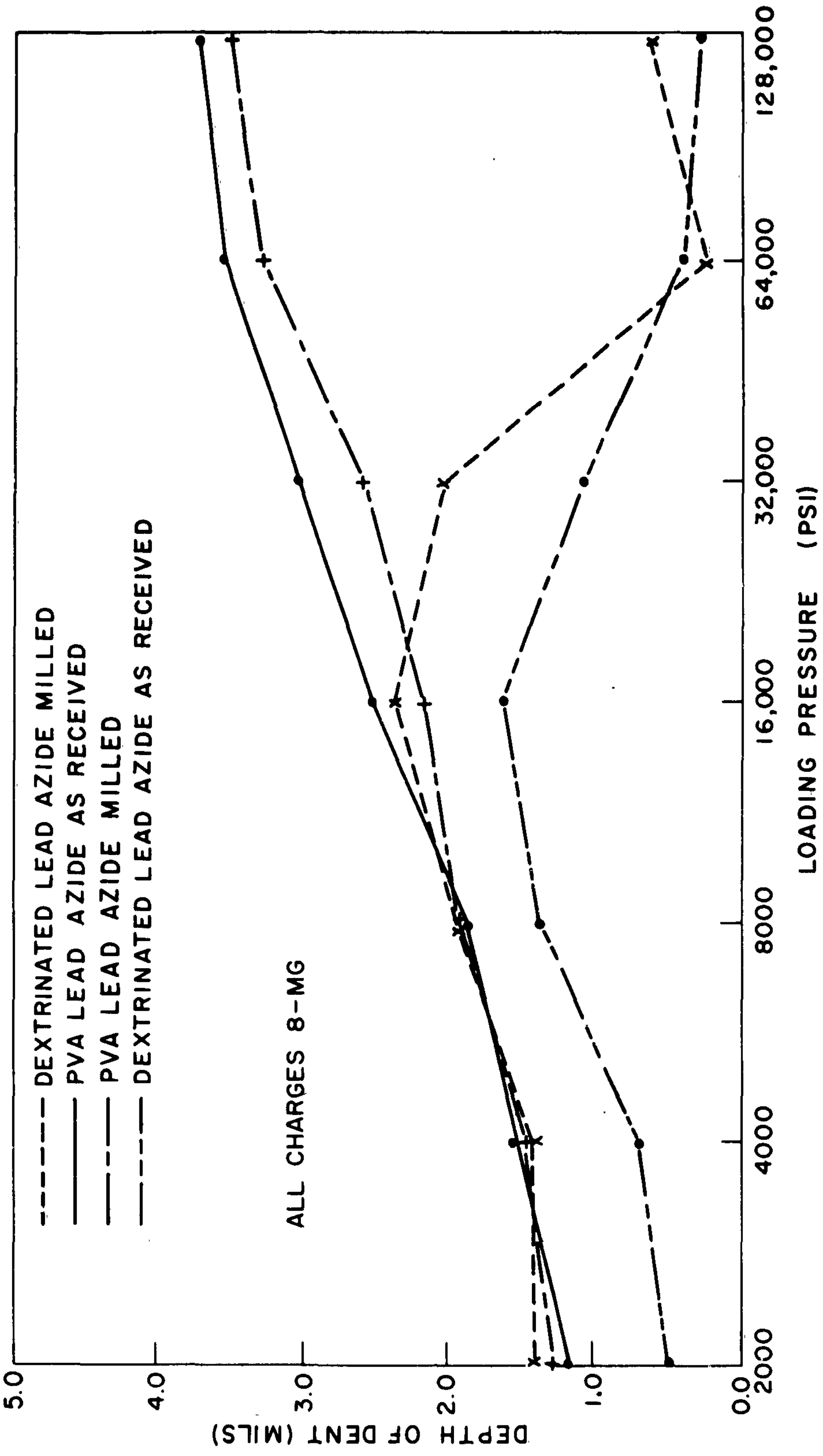

FIG. 5 EFFECT OF MILLING ON DENT OUTPUT OF LEAD AZIDE

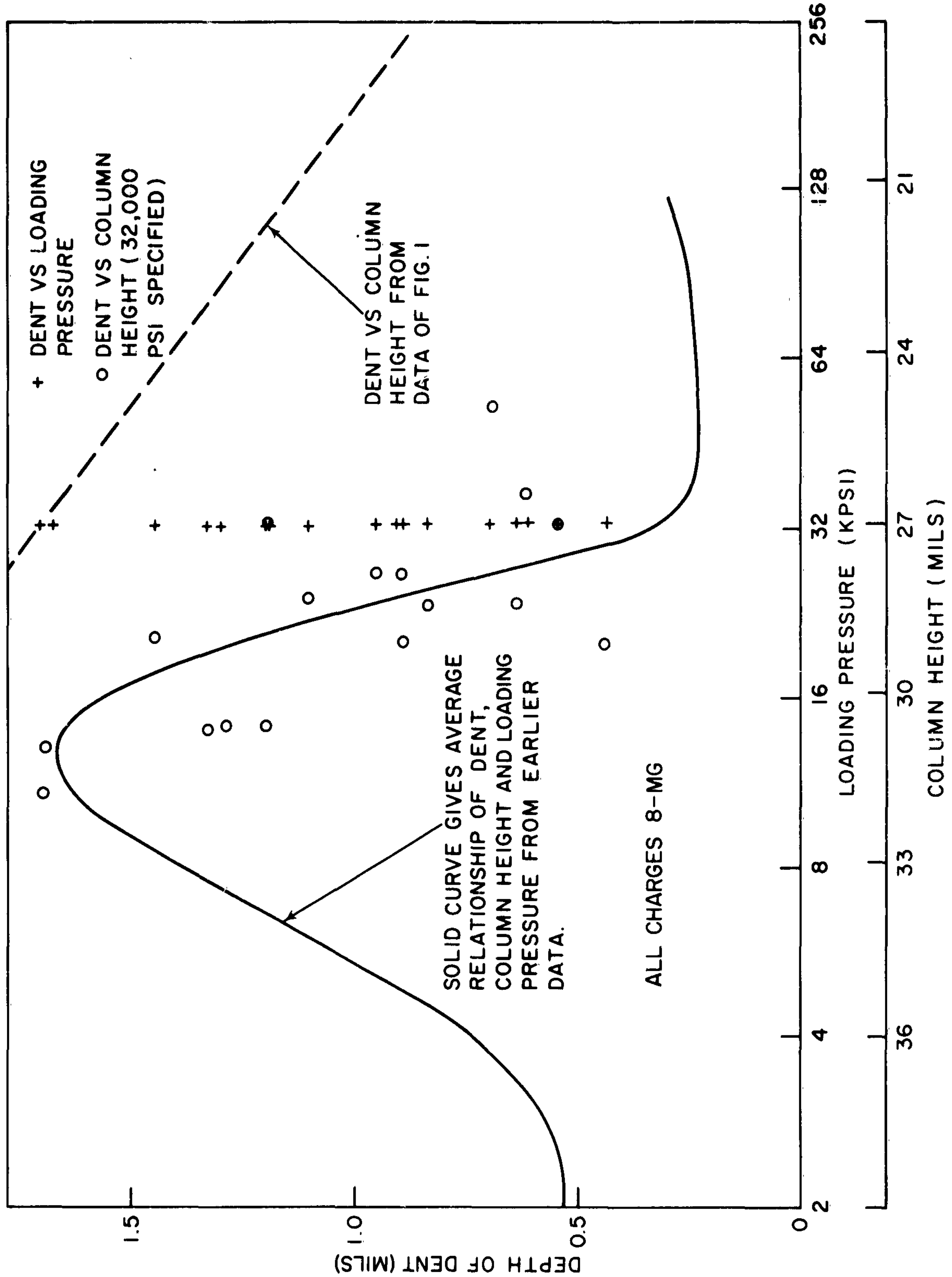

FIG. 6 MICROSCALE DENT TESTS FOR A SINGLE LOT OF DEXTRINATED LEAD AZIDE

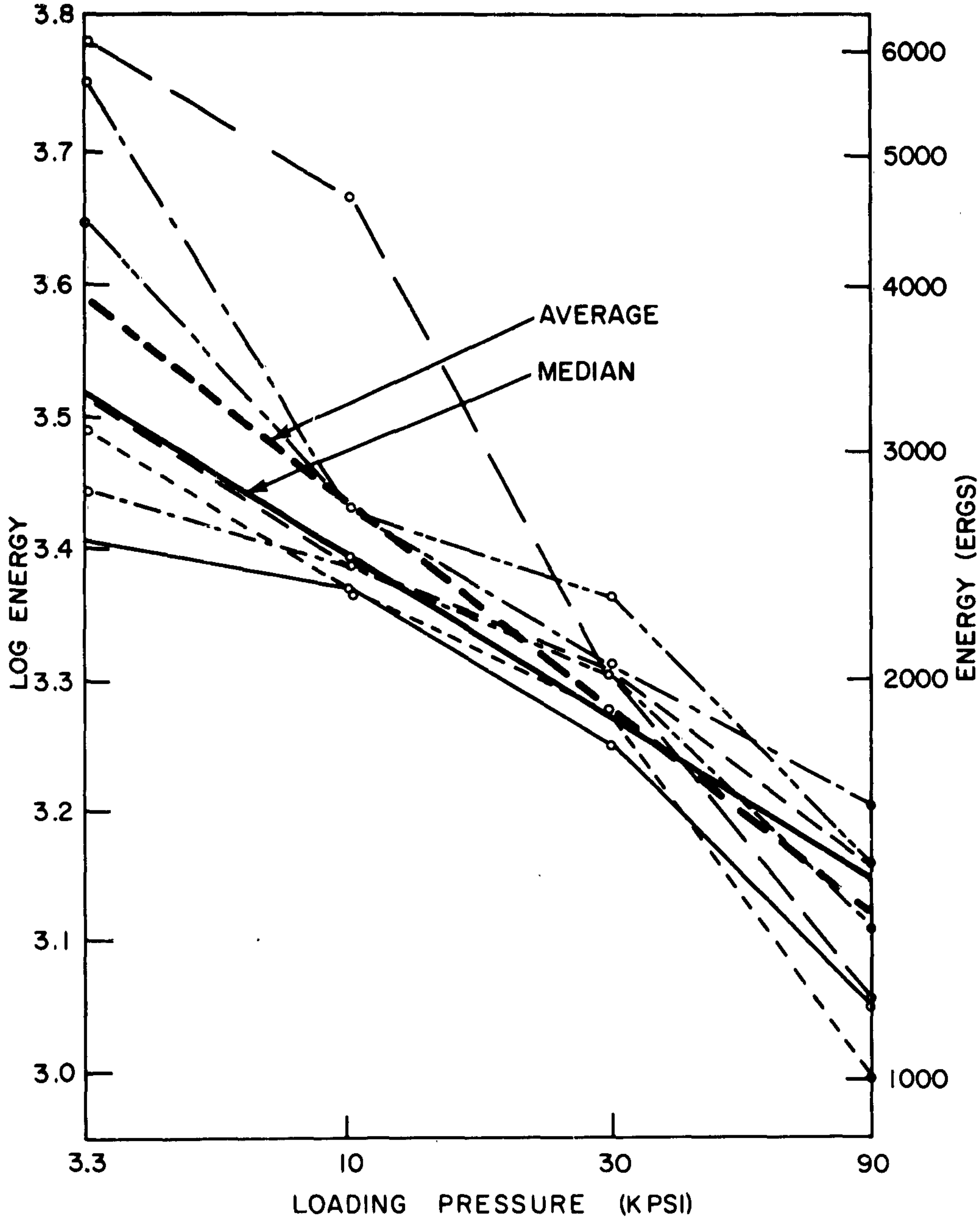

FIG. 7 HOT WIRE SENSITIVITY OF LEAD AZIDE AS A FUNCTION OF LOADING PRESSURE. EACH CURVE REPRESENTS A DIFFERENT ORIGINAL SAMPLE

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OBSERVATIONS OF THE EFFECTS OF LOADING DENSITY ON THE INITIATION AND GROWTH OF DETONATION IN AZIDES, by R.H.F. Stresau and M.H. Rowe. 7 Dec. 1961. 18p. charts, diagr.
UNCLASSIFIED

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1. Lead azides - Detonation
2. Lead azides - Loading
3. Lead azides - Burning rates
4. Lead azides, Dextrinated
5. Wire, Hot - Sensitivity

I. Title
II. Stresau, Richard H.F.
III. Rowe, Marvin H., jt. author

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